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TECHNICAL NOTE

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MEASUREMENT OF TOTAL NORMAL EMITTANCE OF BORON NITRIDE FROM 1,200° F TO 1,900° F WITH NORMAL SPECTRAL EMITTANCE DATA AT 1,400° F

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SUMMARY

A technique basically similar to that of H. O. McMahon was developed for measuring thermal radiation characteristics of ceramic materials. The measurement technique appears to be extremely useful for measuring emittance of nonconductors and materials which are difficult to investigate because of surface-temperature measurement problems. The total emittance values for a 0.253-inch-thick specimen of boron nitride range from 0.79 at 1,210° F to 0.81 at 1,860° F; these values vary approximately linearly with temperature. The total normal emittance value obtained from the spectral emittance data for the 0.253-inch-thick specimen of boron nitride for the wavelength range of 0.5 micron to 15 microns at 1,400° F was 0.75. Specimens of thicknesses ranging from 0.023 inch to 0.253 inch had nearly the same total emittance. The relatively high emittance of boron nitride indicates that this material may be useful as a refractory material for aerodynamic applications.

INTRODUCTION

Boron nitride is a white solid with a high melting point (4,946° F). Its high-temperature properties are such that it holds promise as a refractory material. Boron nitride has been used for rocket-nozzle inserts and is being considered for other high-temperature aerodynamic applications, such as for the leading edges of high-speed aircraft and for thermally critical areas of reentry vehicles.

In order to aid in the evaluation of this material for such applications, a study has been made of its thermal radiation characteristics. The total normal emittance from 1,200° F to 1,900° F of specimens of various thicknesses has been measured in order to determine whether boron nitride was transparent to infrared radiation. The spectral emittance was measured at 1,400° F.

APPARATUS AND TECHNIQUES

The method selected to measure the emittance of boron nitride is based on a technique originally developed in reference 1. In this technique, the specimen is placed in a blackbody cavity constructed of metal; the cavity temperature, measured with thermocouples, is taken as the specimen temperature. This method thus avoids two major problems encountered in the measurement of emittance of insulators such as boron nitride: (1) measuring the surface temperature of the specimen and (2) heating the specimen to a uniform temperature. This method also has the advantage of being applicable to materials that are transparent to infrared radiation.

The measurement of radiant flux from the blackbody is taken with the specimen out of view of the port. The specimen is then moved past the port (fig. 1) and its radiant flux is measured before it can cool significantly.

The furnace, the radiometer for the total normal emittance measurements, and the spectrophotometer for the normal spectral emittance measurements are discussed in the following sections. In addition, the apparatus calibration and the procedure for emittance measurements are discussed.

Furnace

The furnace, shown schematically in figure 1, is based on a design discussed in reference 1. In appearance, it is approximately a 16-inch cube. The unit is constructed of 3,000° F firebrick and is in two parts, an upper half and a lower half, which form a 6-inch cubical cavity in the center when they are placed together.

Each half of the furnace contains a silicon carbide muffle-furnace core which is wound with Kanthal wire. Inside the core is an oxidized Inconel box which minimizes thermal gradients and improves the quality of the blackbody. This box forms the actual blackbody cavity. Temperature measurement is accomplished with four chromel-alumel thermocouples, two on each half of the Inconel box. The temperature of each half of the furnace is controlled separately by 20-ampere autotransformers. The upper operating-temperature limit is approximately 2,100° F.

The lower half of the furnace is equipped with a water-cooled viewing port. The specimen and blackbody are viewed through this port, which is tapered and blackened to minimize reflection of radiation on the specimen during the measurements. The aperture is 0.5 inch in diameter. The specimen, in the form of a semicircle, is mounted perpendicularly on a shaft so that it can be rotated to pass 1/16 inch

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from the viewing port and perpendicular to the axis of the port. There is also in the lower half of the furnace a blackened, water-cooled shutter which may be inserted directly behind the specimen to block any of the blackbody radiation which might be transmitted through a transparent material.

Radiometer

The measurement of radiant flux from the blackbody and from the specimen is accomplished with an industrial radiometer. A schematic diagram of the instrument is shown in figure 2. The basic components consist of a Cassegrainian mirror system, a germanium filter, and a thermistor detector. In order to eliminate radiation in the visible spectrum and the near-infrared spectrum, a germanium filter protects the thermistor detector from radiation that is shorter in wavelength than 1.8 microns. The germanium filter also eliminates wavelengths longer than 25 microns. The electrical output of the detector is amplified and recorded on a fast strip-chart recorder.

Spectrophotometer

The spectral emittance studies were made with a modified spectrophotometer using single-beam operation. Figure 3 includes a schematic diagram of this instrument. Radiation from the furnace enters the spectrophotometer and is chopped at 13 cycles per second. The chopped radiation enters the monochrometer through an entrance slit. In the monochrometer, the radiation is dispersed by a sodium chloride prism in two passes, and radiation of the desired wavelength is positioned on and emerges from the exit slit to be focused on a thermocouple detector. The output of the thermocouple detector is then amplified, rectified, and recorded on a strip-chart recorder.

Apparatus Calibration

Considerable effort was spent in evaluating the apparatus and developing the measuring technique. It was considered desirable to determine whether the specimen temperature and the blackbody temperature were actually close enough to allow the assumption that the specimen temperature and the cavity temperature were the same. In order to determine whether any differential existed, an Inconel specimen with a number of thermocouples attached to its surface was placed in the cavity out of view of the port. The temperature of the specimen and the cavity were compared throughout the temperature range of the furnace and were found to be in close agreement. The results of these measurements are shown in figure 4. The specimen temperatures were obtained by averaging the temperatures of all the thermocouples on the specimen; however, these temperatures differed by less than 1 percent. The furnace temperatures were obtained by averaging the four thermocouple readings, but again the maximum difference in these thermocouple readings was less than 1 percent. The specimen temperature was consistently below that of the furnace; the maximum difference was 30° F at $1,900^{\circ}$ F, the highest experimental temperature. This deviation is 1.3 percent of the absolute temperature. This corresponds to an error in emittance values of 5 percent to 6 percent. These measurements indicate that the basic assumption that the specimen was at the same temperature as the furnace was valid for the nonmeasuring portion of the cycle and that thermal equilibrium was established between the specimen and the blackbody cavity. Since calibrated thermocouples were not used and the accuracy of the temperature-recording instruments was only 0.5 percent of the reading, no data corrections were made for the temperature differences.

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When the specimen is rotated in front of the viewing port, it radiates to the cooler surroundings and obviously must cool. Therefore, the effect that cooling during the viewing cycle might have on the experimental results had to be determined. In order to accomplish this, the following approach was taken. The thinnest specimen (0.023 inch) of boron nitride was placed in the furnace out of sight of the viewing port and heated to $1,900^{\circ}$ F, the highest experimental temperature. When the furnace temperature had stabilized, radiant-flux measurements were made in the following manner. With the specimen away from the viewing port, the blackbody radiant flux was measured. The motor-driven shaft then rotated the specimen at 15 rpm. At the first sight of the specimen at the viewing port, the water-cooled shutter was inserted behind the specimen and the radiant flux from the specimen was measured. When the specimen was away from the port, the shutter was withdrawn. The flux from the specimen was measured in this manner for 12 consecutive cycles and recorded on a strip-chart recorder. The result of this test is shown in figure 5. The first measurement is called the 1/2 cycle because the specimen had been subjected to 1/2 cycle of cooling. By comparing the ratio of the flux from the specimen to the flux from the blackbody for each cycle, it is possible to determine the effect of cooling on the emittance values. The emittance measured for the 1/2 rotation cycle of the specimen was 0.70; that measured for the $1\frac{1}{2}$ rotation cycle was 0.67. This difference of 4 percent is discussed in the section entitled "Errors."

Procedure for Emittance Measurements

The procedure outlined in the following descriptions was adopted as the method which gave the most consistent and reliable data for both the spectral and the total normal emittance measurements.

Total normal emittance.- For measuring total normal emittance, the specimen was positioned on the rotating shaft in a manner to allow it to pass 1/16 inch from the viewing port during the measuring cycle. With the specimen in position, the radiometer was positioned and focused on the specimen. The furnace halves were placed together and power was applied to the heater windings through the two autotransformers. The temperature of the Inconel liner in each half of the furnace was monitored, and corrections were made by manually regulating the autotransformers to eliminate any temperature differential. When the furnace had reached the desired temperature and had stabilized, the output of the radiometer was recorded on a strip-chart recorder. This value was taken as corresponding to blackbody flux at the measured furnace temperature. The shaft motor was turned on. The water-cooled shutter was inserted behind the specimen as it rotated past the viewing port, and the radiometer output was recorded on a strip-chart recorder. This value was taken as corresponding to specimen flux at the measured furnace temperature. The specimen was then stopped out of sight of the port and allowed to remain there for 5 minutes. This procedure was then repeated in order to determine whether any changes in emittance were taking place as a result of the high-temperature environment of the specimen. No changes were found. The values of total normal emittance were obtained from the following expression:

$$\text{Emittance} = \frac{\text{Radiometer output for specimen radiation (rms volts)}}{\text{Radiometer output for blackbody radiation (rms volts)}}$$

Spectral emittance.- The specimen positioning and heating procedures for the spectral emittance measurements were identical to those used for the total emittance measurements. The radiometer was replaced with the modified spectrophotometer. The furnace was positioned to allow the monochrometer slit to be completely filled with radiation from the viewing port. With the furnace at the desired temperature, the blackbody radiant output at 0.5 micron was recorded; then the specimen, with the water-cooled shutter behind it, was viewed at the same wavelength, and its output was recorded. The ratio of the radiant output from the specimen to the radiant output from the blackbody gives the value of emittance at that particular wavelength. This procedure was repeated at 0.5-micron intervals up to 15 microns. The width of the slit was changed at each wavelength interval in order to obtain nearly the maximum recorder deflection, thus minimizing errors in calculating emittance from chart records.

SPECIMENS AND TESTS

Specimens of various thicknesses were machined from 4-inch-diameter pressed boron nitride round stock in the form of a semicircle. The initial specimens were carefully polished on decreasing grits of emery paper to insure a uniform surface. After polishing, the specimens were dried at 212° F to remove any absorbed water and the thicknesses were measured with a micrometer. Subsequent measurements of emittance of the polished and "as machined" specimens indicated, however, that the various surface preparations produced no detectable changes in emittance, probably because the "as machined" specimens had a relatively smooth surface.

The total normal emittance of specimens of boron nitride of thicknesses of 0.023 inch, 0.029 inch, 0.044 inch, 0.062 inch, 0.072 inch, 0.129 inch, 0.197 inch, and 0.253 inch was measured from $1{,}200^{\circ}$ F to $1{,}900^{\circ}$ F.

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The normal spectral emittance of a 0.253-inch-thick specimen of boron nitride at $1{,}400^{\circ}$ F in the wavelength range of 0.5 micron to 15 microns was measured.

ERRORS

The following discussion summarizes the errors that can be expected in the data due to (1) the germanium filter, (2) the specimen temperature being lower than that of the blackbody, and (3) the cooling of the specimen as it passes the port.

Since the radiometer used for the total normal emittance measurements has a germanium filter which eliminates wavelengths shorter than 1.8 microns and longer than 25 microns, a certain percentage of the energy will be lost in the total normal emittance measurements. According to Wein's displacement law, the maximum error due to the 1.8-micron cutoff will occur at the highest experimental temperature, $1{,}900^{\circ}$ F. At $1{,}900^{\circ}$ F for a blackbody 14 percent of the energy is below 1.8 microns and hence will not be taken into account in the total normal emittance measurements. The maximum error due to the 25-micron cutoff of the filter will occur at $1{,}200^{\circ}$ F, but here less than 1 percent of a blackbody energy is above 25 microns. No data corrections were made for this error since the spectral distribution of boron nitride in the wavelength range below 1.8 microns is not known; hence, a correction could not accurately be made, but the maximum error will be at $1{,}900^{\circ}$ F and it will be less than 14 percent.

The error caused by the specimen temperature being 1.3 percent lower than the temperature of the blackbody at $1{,}900^{\circ}$ F will cause an error of 5 percent to 6 percent in emittance values. The effect of the specimen temperature will not be as great as 6 percent, however, since the emittance of the blackbody is less than unity.

Cooling the specimen during rotation caused a decrease of 4 percent in emittance values between the first and second passes of the specimen by the viewing port. Since the specimen was subjected to some cooling before the first measurement was taken, an approximate calculation was made of this cooling as the specimen passed the port. It was assumed that the specimen was radiating to two surfaces at 70° F and the temperature decrease was calculated. This decrease was 5 percent of the absolute temperature and would cause an error in emittance values of approximately 20 percent. This 20-percent error represents the maximum possible error that can be expected in the data as a result of the cooling of the specimen.

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It should be pointed out that in the spectral emittance data, although the resolution of the instrument decreases below 1 micron, values were taken at a wavelength of 0.5 micron.

RESULTS AND DISCUSSION

Figure 6 shows the total normal emittance values as a function of temperature from 1,200° F to 1,900° F for specimens of boron nitride of eight different thicknesses varying from 0.023 inch to 0.253 inch. The total normal emittance values for the thinnest specimen of boron nitride (0.023 inch) range from 0.72 at 1,270° F to 0.74 at 1,875° F. The emittance values for the thickest specimen (0.253 inch) range from 0.79 at 1,210° F to 0.81 at 1,860° F; the variation is approximately linear with temperature. Specimens of various thicknesses ranging from 0.023 inch to 0.253 inch had nearly the same total emittance values.

In order to determine whether boron nitride was slightly transparent to infrared radiation, plots of measured emittance against specimen thickness varying from 0.023 inch to 0.253 inch were made for several temperatures. These curves were obtained by drawing the best straight line through the points on the curves of emittance plotted against temperature (fig. 6) and taking points from this line at 1,400° F, 1,700° F, and 1,900° F. Figure 7 shows these plots. These plots appear to show an increase in emittance with specimen thickness as would be expected for a material that is transparent to infrared radiation. Because of the difference in the cooling of the thick and thin specimens and the scatter of the data, it is believed that this effect cannot be accurately estimated.

Figure 8 shows the data for two spectral emittance tests for a 0.253-inch-thick specimen of boron nitride from 0.5 micron to 15 microns at a temperature of 1,400° F. The emittance is about 0.15 at 0.5 micron. The emittance increases to a maximum of 0.95 at 6.0 microns and then drops sharply to 0.62 at 6.5 microns. The emittance increases again and is between 0.83 and 0.93 from 8.5 microns to 15 microns. The curve faired through the data points is in good agreement with the spectral emittance data for boron nitride at a slightly different temperature as reported in reference 2. The spectral emittance values measured in the two tests differed by a maximum of ±3 percent at any particular wavelength; however, the difference was much less over the most important parts of the curve. The integrated spectral emittance, obtained from the expression

$$\text{Spectral emittance} = \frac{\int_{0.5}^{15} (\text{Specimen energy})d\lambda}{\int_{0.5}^{15} (\text{Blackbody energy})d\lambda}$$

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(where λ represents wavelength) was 0.75, which is in good agreement with the value 0.79 for total normal emittance measured with the radiometer for the same specimen at 1,400° F but for the wavelength range of 1.8 microns to 25 microns.

CONCLUDING REMARKS

A technique was developed for measuring thermal radiation characteristics of ceramic materials. The measurement technique appears to be extremely useful for measuring emittance of nonconductors and materials which are difficult to investigate because of surface-temperature measurement problems. The total emittance values for a 0.253-inch-thick specimen of boron nitride range from 0.79 at 1,210° F to 0.81 at 1,860° F; these values vary approximately linearly with temperature. The total normal emittance value obtained from the spectral emittance data for the 0.253-inch-thick specimen of boron nitride for the wavelength range of 0.5 micron to 15 microns at 1,400° F was 0.75. Specimens of thicknesses ranging from 0.023 inch to 0.253 inch had nearly the same total emittance. The relatively high emittance of boron nitride indicates that this material may be useful as a refractory material for aerodynamic applications.

Langley Research Center,
National Aeronautics and Space Administration,
Langley Station, Hampton, Va., March 15, 1962.

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2. Blau, Henry H., Jr., Marsh, John B., Martin, William S., Jasperse, John R., and Chaffee, Eleanor: Infrared Spectral Emittance Properties of Solid Materials. AFCRL-TR-60-416, Geophys. Res. Directorate, Air Force Res. Div., Oct. 1960.

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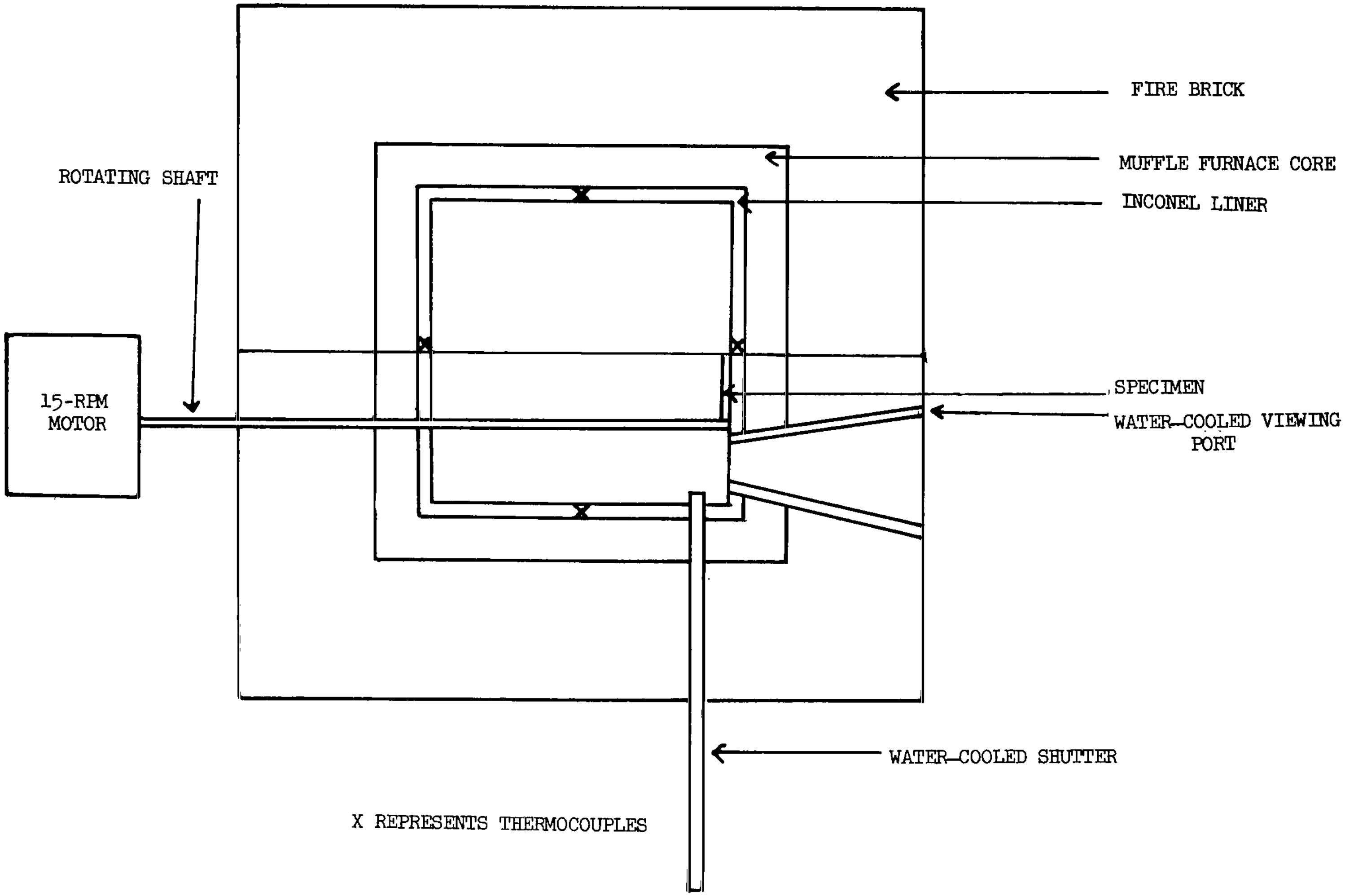

Figure 1.- Schematic diagram of blackbody furnace for total normal emittance measurements.

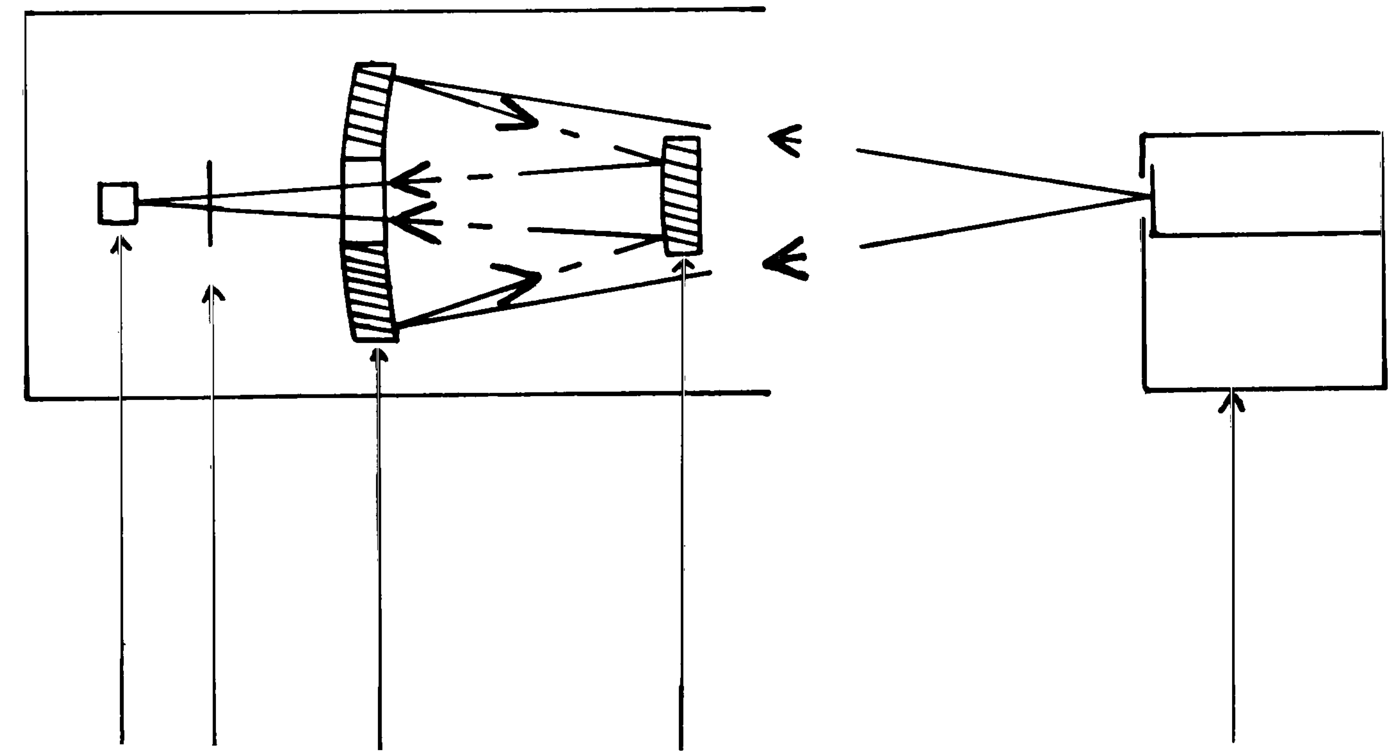

Figure 2.- Schematic diagram of apparatus for total normal emittance measurements.

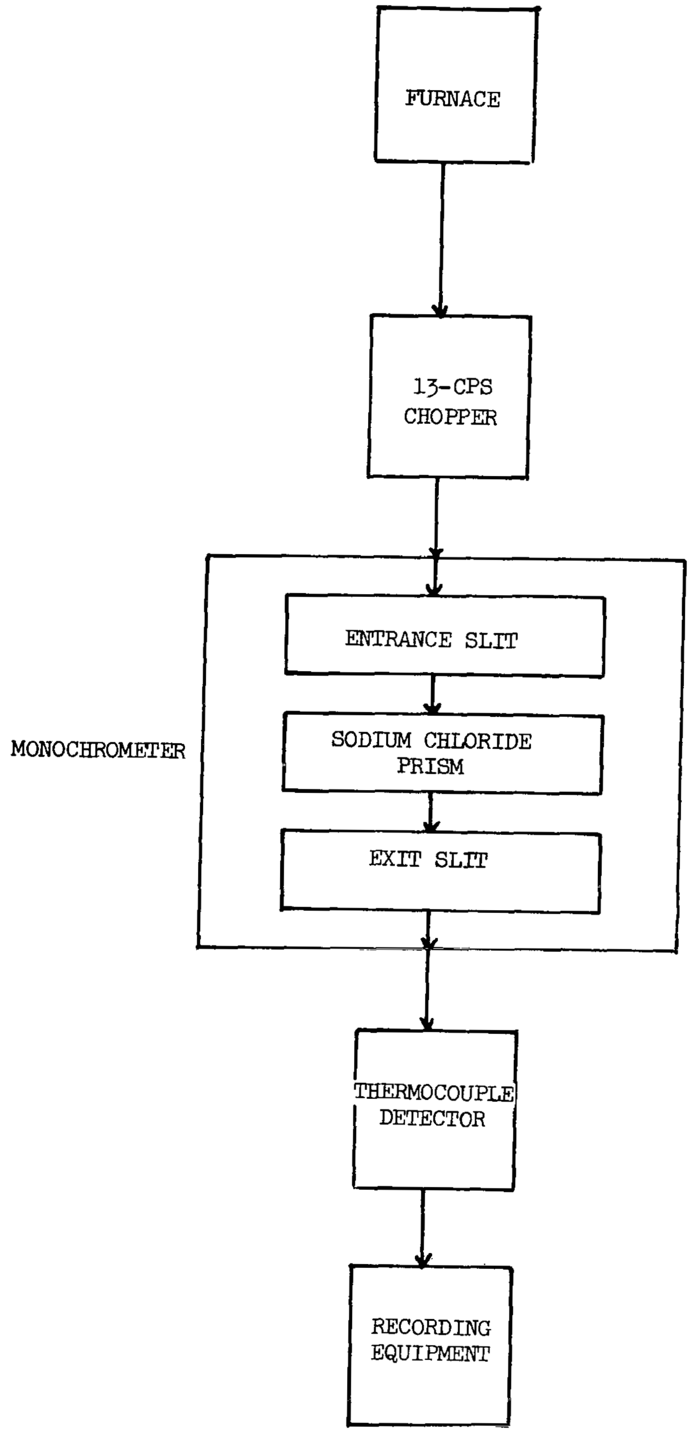

Figure 3.- Schematic diagram of apparatus for spectral emittance measurements.

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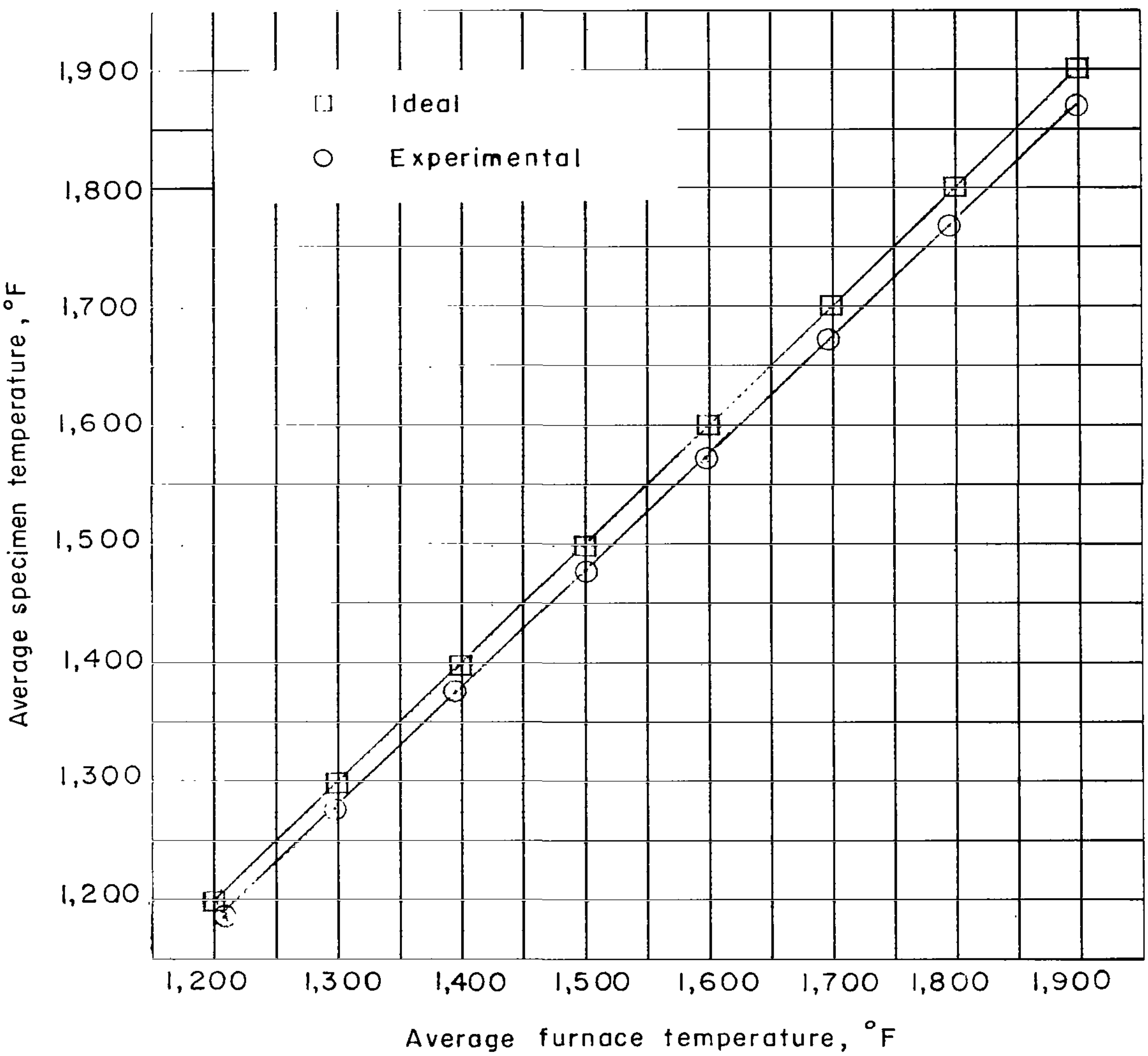

Figure 4.- Comparison of specimen and furnace temperature.

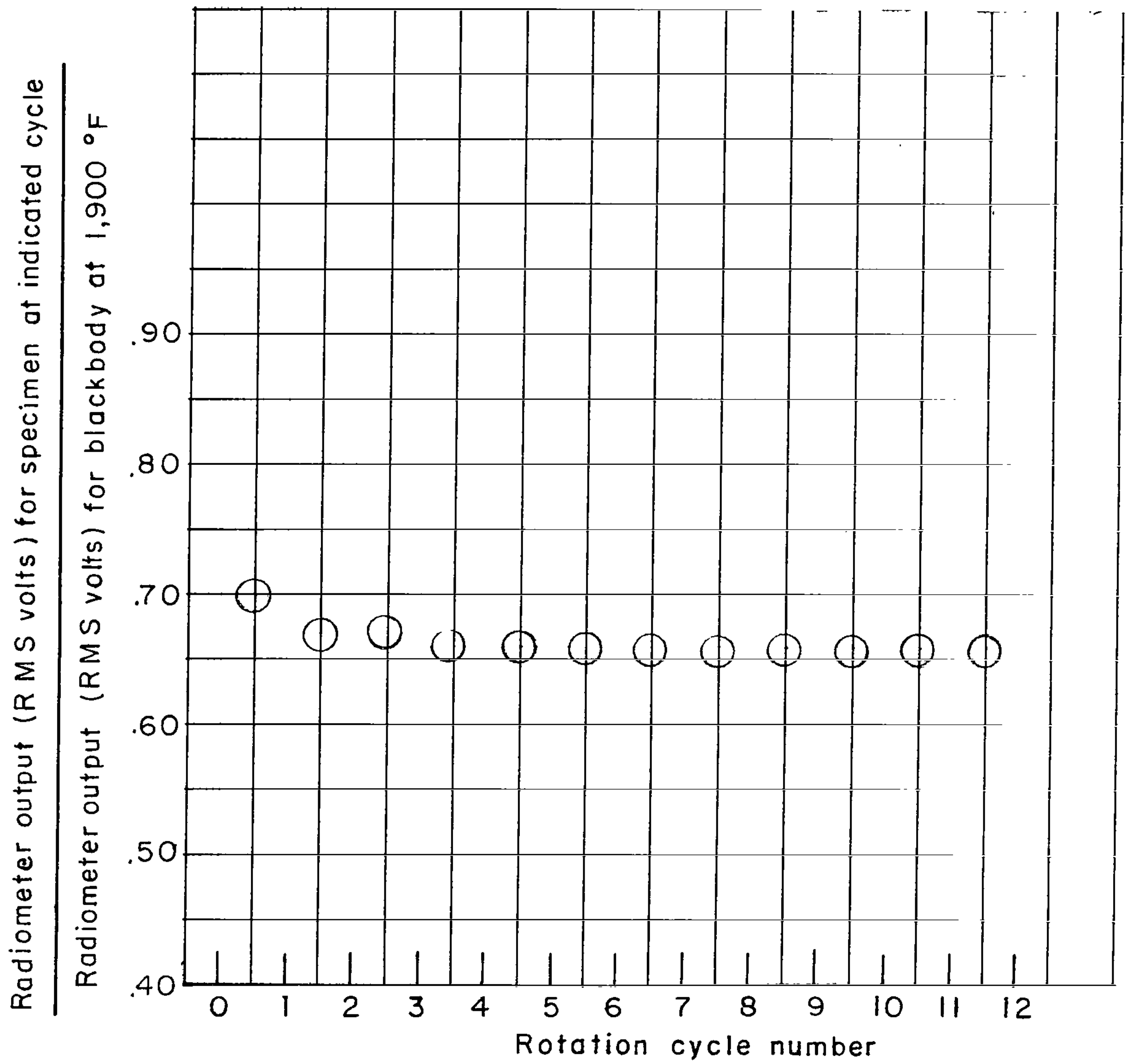

Figure 5.- Decrease of radiant intensity ratio for a 0.023-inch-thick specimen of boron nitride rotated for consecutive cycles at a furnace temperature of 1,900° F.

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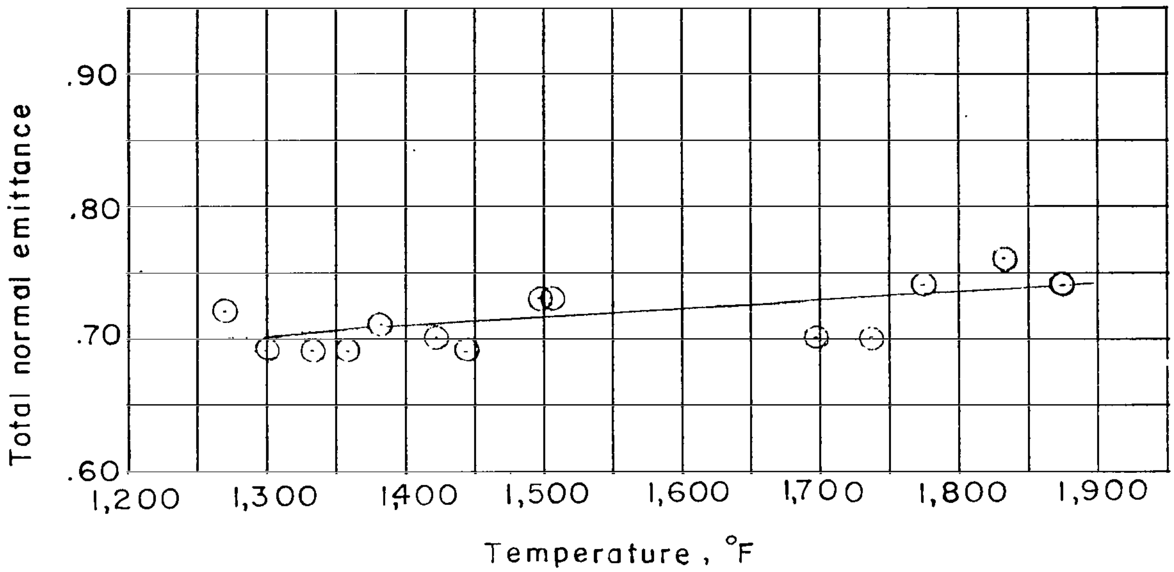
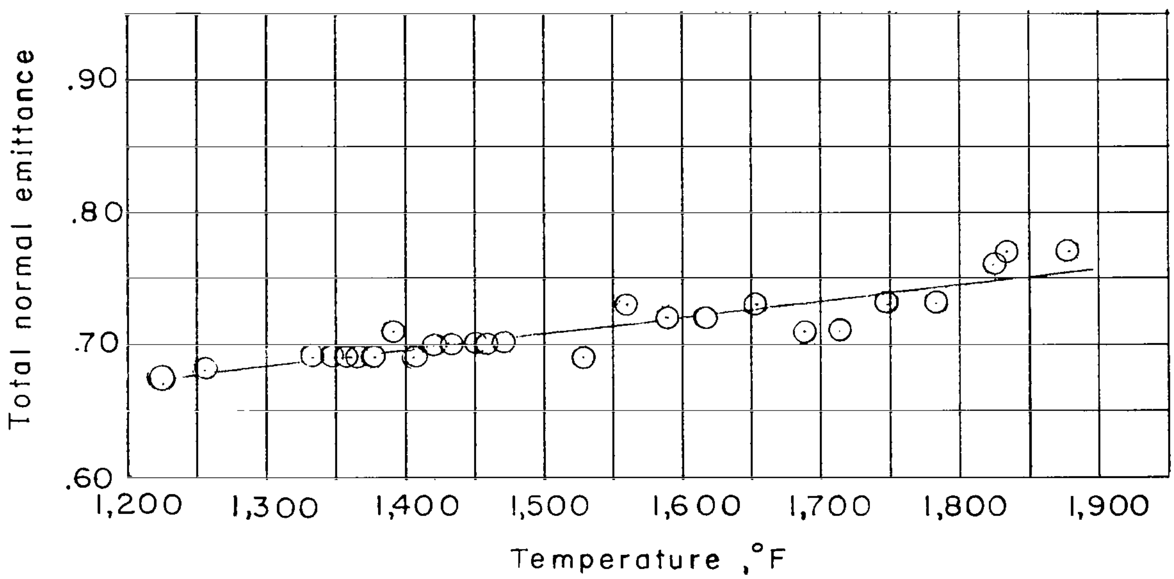
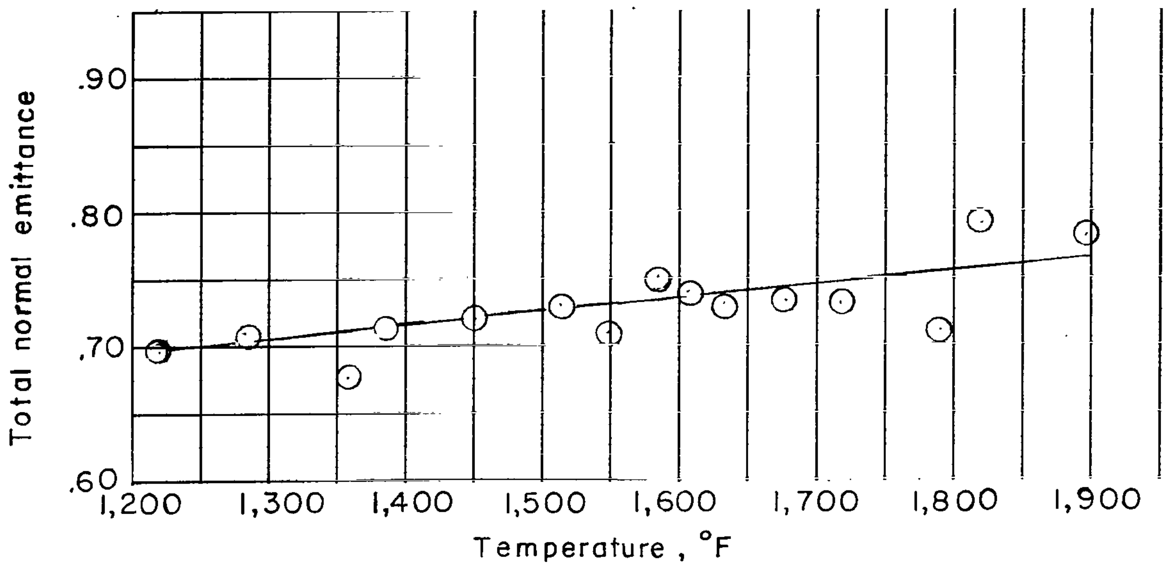


(a) 0.023 inch.

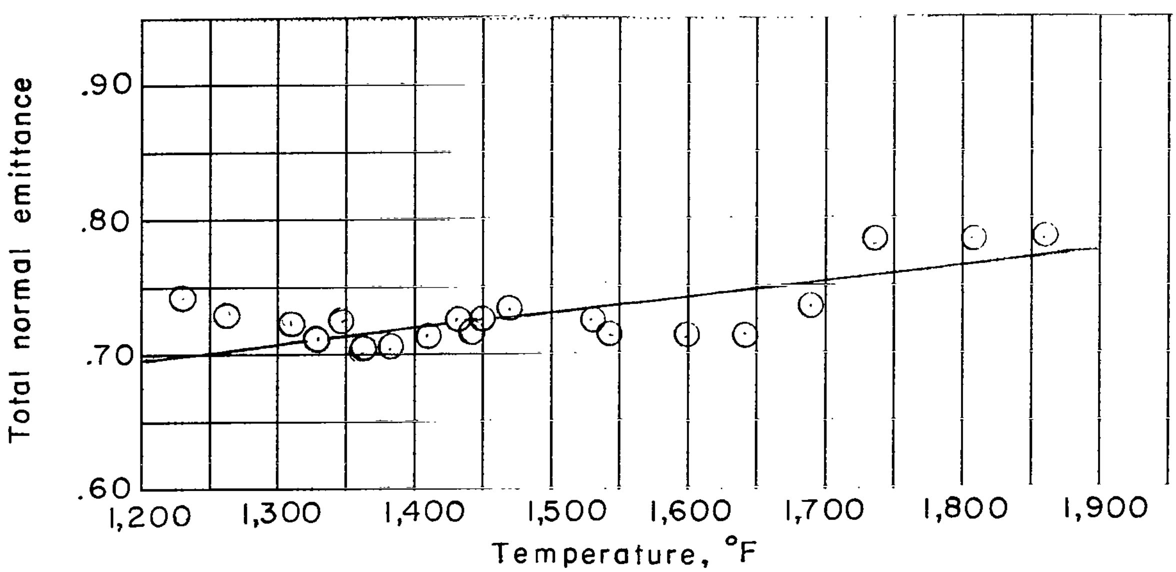


(b) 0.029 inch.

Figure 6.- Total normal emittance as a function of temperature for specimens of boron nitride various thickness.



(c) 0.044 inch.



(d) 0.062 inch.

Figure 6.- Continued.

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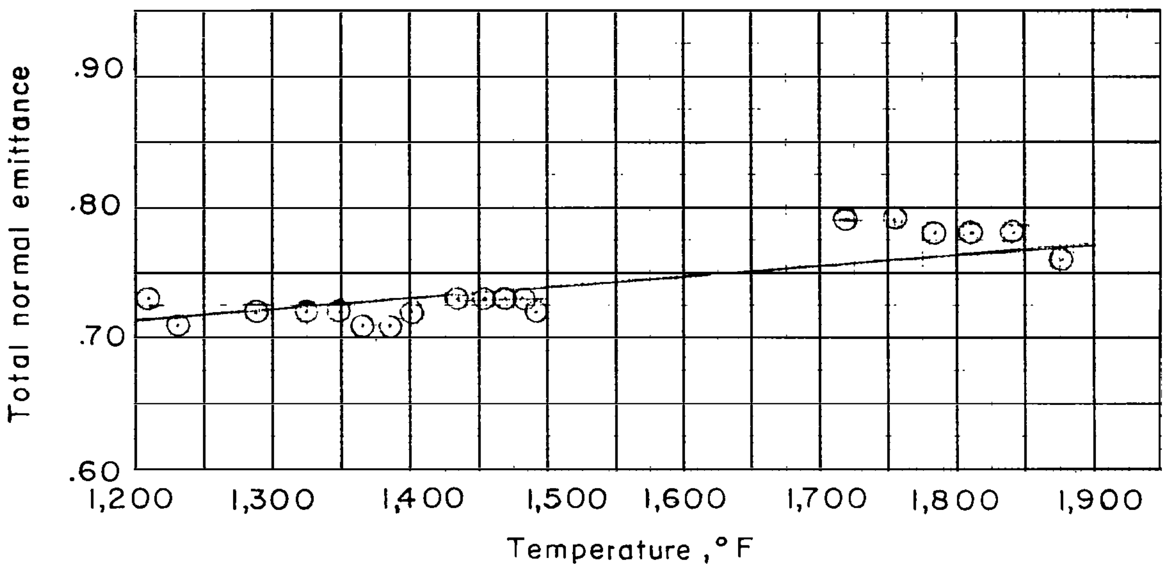
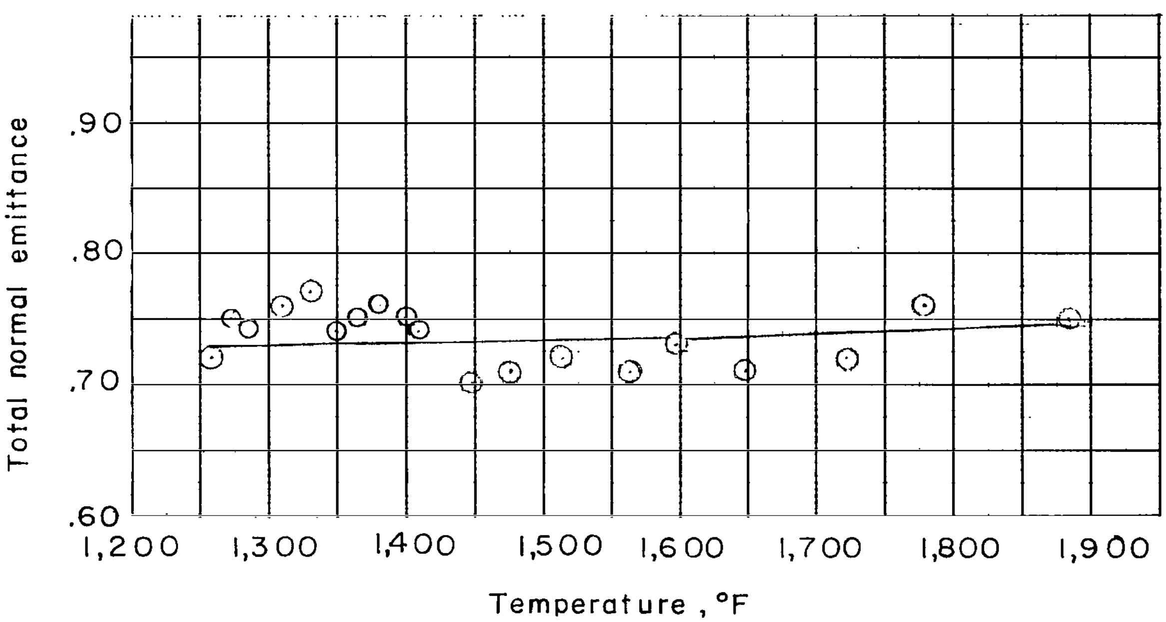
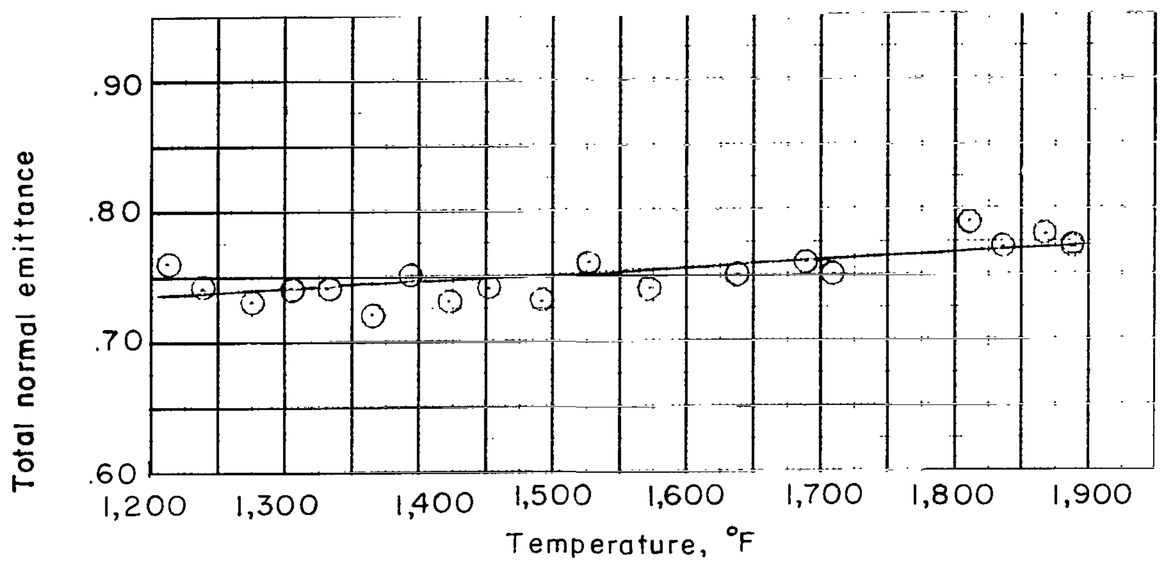


(e) 0.072 inch.

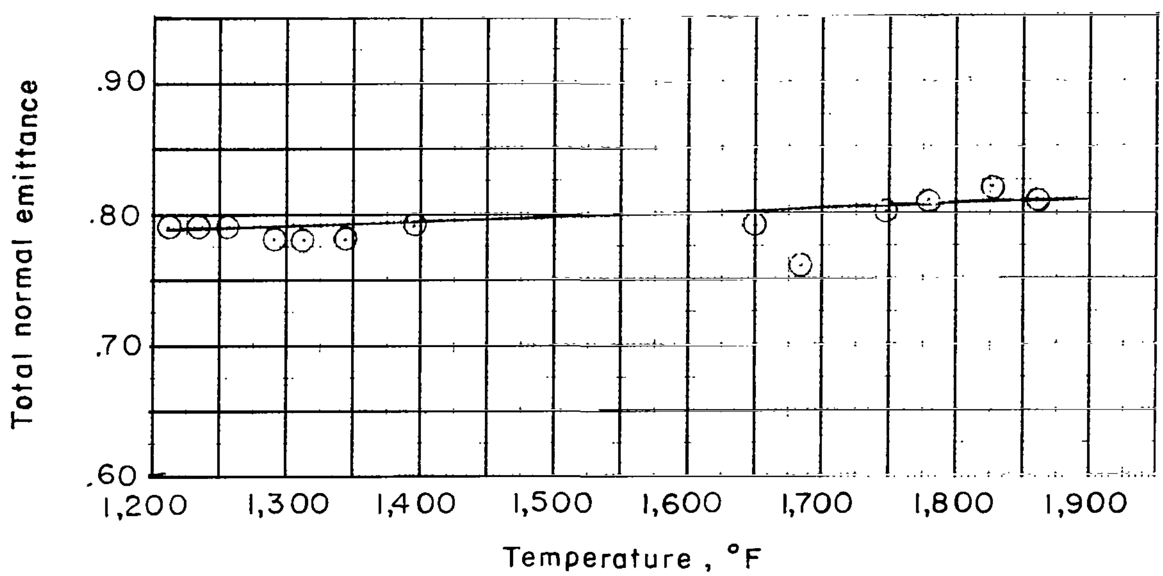


(f) 0.129 inch.

Figure 6.- Continued.



(g) 0.197 inch.



(h) 0.253 inch.

Figure 6.- Concluded.

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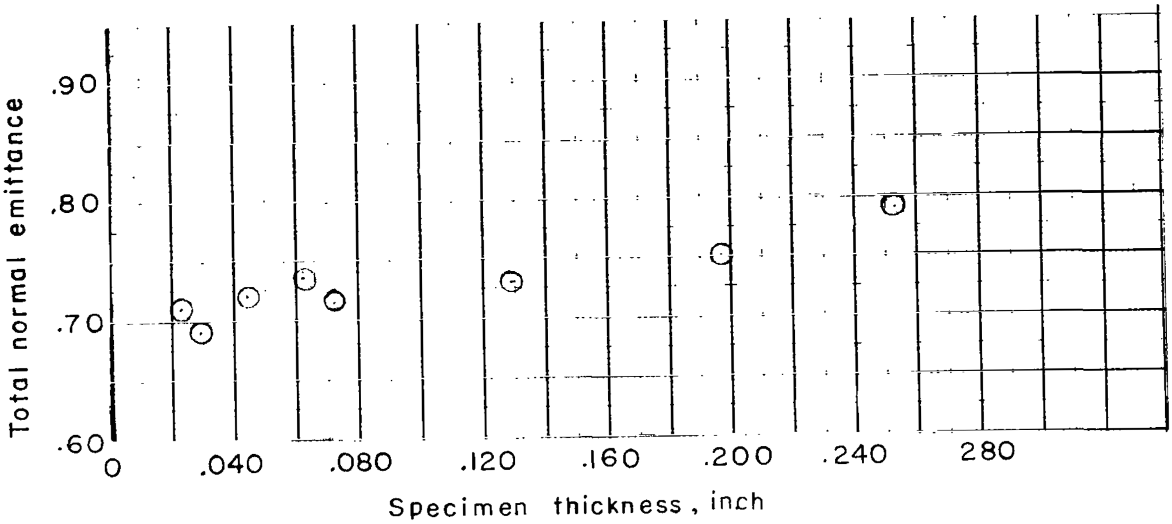
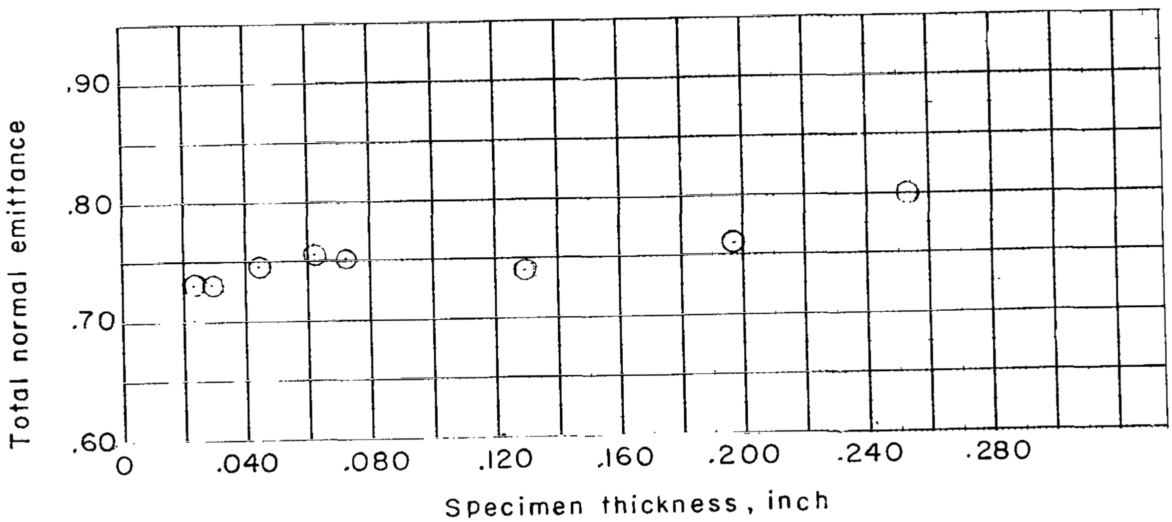
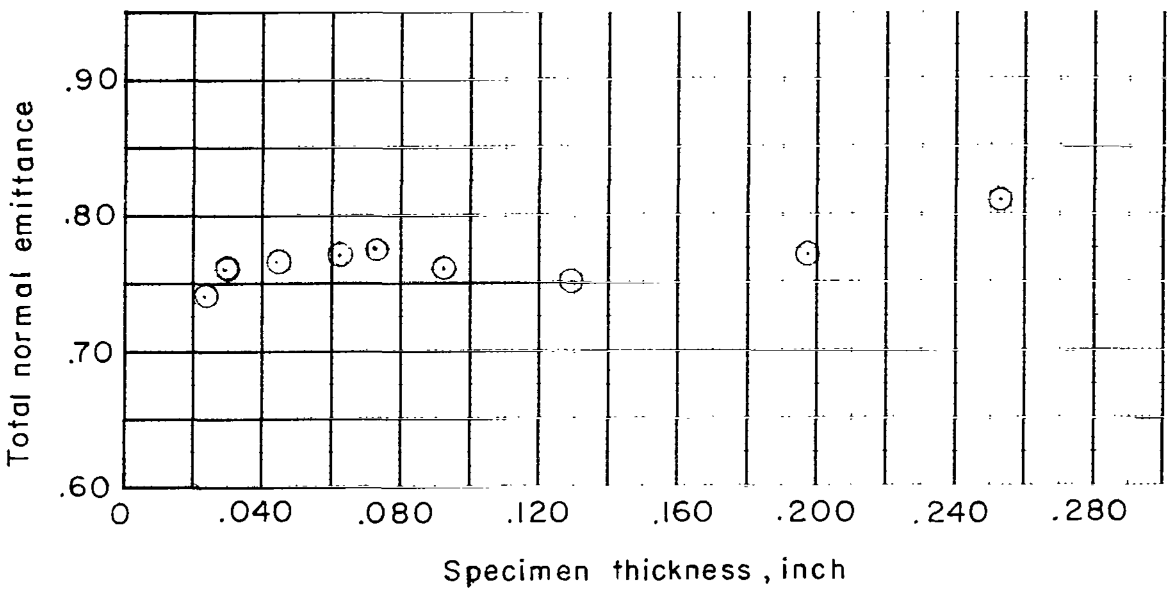


(a) 1,400° F.



(b) 1,700° F.

Figure 7.- Total normal emittance as a function of specimen thickness



(c) 1,900° F.

Figure 7.- Concluded.

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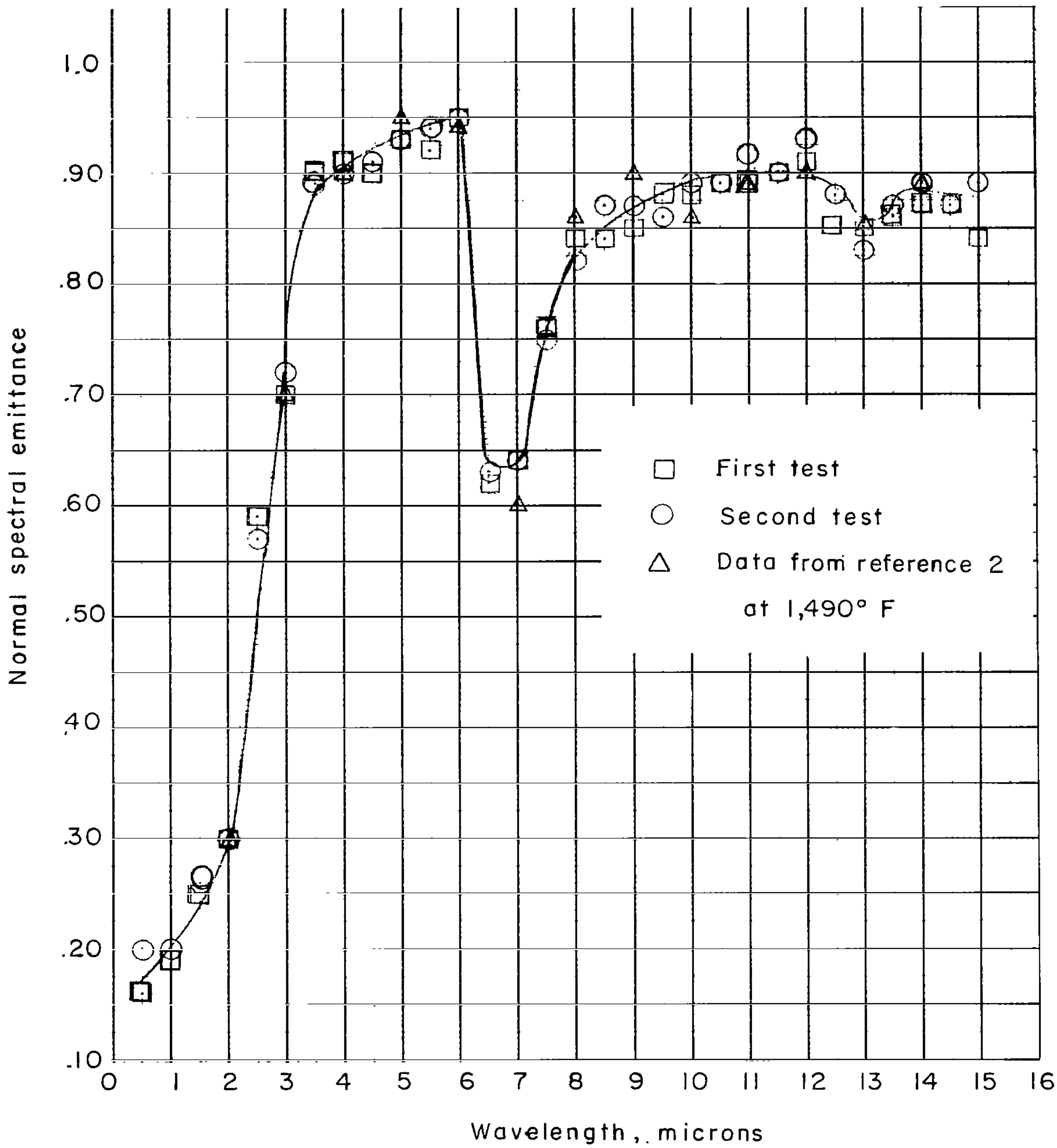

Figure 8.- Normal spectral emittance of 0.253-inch-thick specimen of boron nitride at a temperature of 1,400° F.